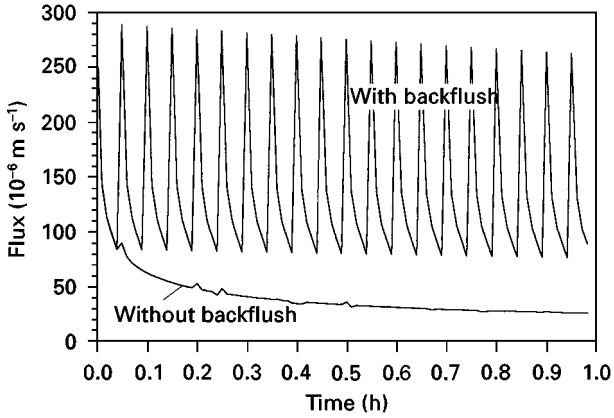

Figure 12 Microfiltration flux when filtering a particle suspension, with and without backflush.

(about 0.5 bar) for several seconds every few minutes (**Figure 12**).

Fouling is reduced by high cross-flow velocities and low transmembrane pressures. High cross-flow velocities cause high-pressure drops along the membrane, which cause the ΔP to be undesirably high at the entrance of the membrane module. Therefore microfiltration processes have been developed which facilitate a cross-flow both on the feed side and on the permeate side. The pressure drops on both sides are similar in magnitude, guaranteeing a *uniform transmembrane pressure*. This method of operation has been shown to be effective in many dairy applications.

Other process techniques to reduce fouling are the use of pulsed flow, gas sparging, and electric or acoustic fields, and the use of flow geometries that create secondary flows or vortices resulting in high shear rates (e.g. the use of 'turbulence promoters' or curved channels).

Conclusions

Over the last 70 years, microfiltration has developed from a small specialized technology used only in laboratories to a multibillion dollar industry for separation and purification of liquid and gas streams. Especially since the 1980s, exciting new applications have become possible, due to improved membranes (for example, ceramics) and improved technologies (for example, backpulsing, uniform transmembrane pressure). Still, great challenges exist, for example in the processing of beverages, such as fruit juices, milk, and beer, where membrane fouling seriously impairs the economy of the process.

To overcome these problems, researchers and engineers are becoming increasingly interested in hybrid and combined processes. Combining microfiltration with good pre- and post-treatments or with other separation processes may result in better and more economic separations.

See also: **II/Membrane Separations:** Filtration.

Further Reading

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Pervaporation

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Development

In 1917 PA Kober published a paper in which he described his observation that 'a liquid in a collodion bag, which was suspended in the air, evaporated, although the bag was tightly closed'. Kober was not the first researcher to observe this phenomenon, but the first to realize its potential for the separation of liquid mixtures which otherwise are difficult to separate, and to separate them under moderate conditions. He introduced the terms 'Pervaporation', and 'Perstillation', and the first term is now in use to describe in

general a process in which one component out of a fluid mixture selectively permeates through a dense membrane, driven by a gradient in partial vapour pressure, leaving the membrane as a vapour, and being recovered in a condensed form as a liquid.

In the years following Kober's publication a number of papers were published describing membranes and processes for pervaporation. Especially during the 1950s, the interest focused on pervaporation membranes and processes for the separation of different classes of hydrocarbons and of isomeres and numerous patents were granted. Membrane materials disclosed were natural and synthetic rubbers, cellulose esters and ethers, and several treated and untreated polyolefines. None of this early membrane, however, was used in any industrial process, owing to insufficient flux and selectivity.

Pervaporation, vapour permeation and gas permeation are very closely related processes. The driving force is always a gradient in partial vapour pressure, and transport through the membrane can best be described by a so-called 'Solution-Diffusion-Mechanism'. In this mechanism it is assumed that a component of the feed having a high affinity to the membrane is easily and preferentially absorbed and dissolved in the dense membrane. Following a concentration gradient it migrates through the membrane by a diffusion process and is desorbed at the downstream side of the membrane. The separation characteristic of the membrane is thus governed primarily by the solubility of components in the membrane material and, to a lesser extent, by its diffusivity which even may counteract against the solubility separation.

In pervaporation and vapour permeation processes the partial vapour pressures of the components at the feed side are fixed by composition and temperature of the feed; they can be influenced only by increasing the temperature. Therefore, the driving force for the transport of matter through the membrane is applied by reducing the partial vapour pressure at the permeate side.

Different means have been proposed in order to effect this reduction of the permeate side partial vapour pressure:

- The permeate side of the membrane is swept with an inert gas in which the partial vapour pressure of the critical (preferential permeating) component is kept sufficiently low. If the gas stream cannot be wasted it has to be reconditioned and recycled.
- All permeating vapour is removed by means of a vacuum pump. The vapour may be condensed after recompression at the downstream side of the pump.
- The permeated vapour is condensed at sufficiently low temperatures. As the condenser surface will be installed at a certain distance to the permeate side of the membrane all non-condensable gases have to be removed from the permeate compartment in order to minimize permeate side pressure losses.

In most industrial installations the last has been proven to be the most effective and economical process.

In a pervaporation process the feed is applied as a liquid and all partial vapour pressures of the components in the feed mixture are at saturation level. Within the limits of membrane stability and process requirements, temperature and pressure on the feed side are free adjustable parameters.

In vapour permeation a vaporous feed mixture is applied, with at least the partial vapour pressure of the preferential permeating component at or close to saturation conditions. Temperature and pressure of the feed are linked by vapour–liquid equilibrium and can be chosen within these limits only.

In gas permeation all partial vapour pressures at the feed side are below saturation and the permeate can no longer be condensed. By increasing the total feed side pressure the driving force for the transmembrane transport can be adjusted.

Pervaporation treatment liquid feed mixtures is insofar unique compared with other membrane processes as the transport of matter across the membrane is coupled with a phase change from liquid to vapour. The heat of evaporation is extracted from the liquid feed and transported through the membrane, too. As a consequence the temperature of the feed is reduced, which reduces driving force and transmembrane flux. Different means such as heated modules have been proposed to replace the lost heat of evaporation. In general, the total membrane area is split into a number of segments (stages) arranged in series with intermediate heat exchangers between each two segments or stages.

Membranes and Modules

Membranes

With the much broader knowledge of membrane structure and membrane manufacture accumulated in the development of desalination membranes in the 1970s pervaporation processes gained new interest. The separation characteristic of a membrane process is determined by the difference in transport rates of the components through the membrane only, not by liquid–vapour equilibria, and azeotropic mixtures can easily be separated. Since only the heat of

evaporation of the permeate vapour is lost a single step membrane process saves energy compared with, e.g., distillation.

Two different types of pervaporation membranes were developed at about the same time in the beginning of the 1980s:

- Hydrophilic membranes, with a preferential permeation for water, used mainly for the removal of water from organic solvents and solvent mixtures, with an emphasis on azeotropic mixtures.
- Organophilic membranes for the removal of volatile organic components from water and gas streams.

In both applications composite membranes are used, allowing for very thin separation layers but with sufficient chemical, mechanical and thermal stability. Because the composite structure flat sheet configurations are preferred. The substructure of both types of pervaporation membranes is very similar: a porous support membrane with an asymmetric pore structure is laid onto a carrier layer of a woven or nonwoven textile fabric and a basic ultrafiltration membrane is formed. On the free side of this porous substructure the pores have diameters in the order of 20–50 nm which widen up to the fabric side to the micrometre range. Polyester, polypropylene and similar fibres are used for the textile carrier layer; structural polymers such as polyacrylonitrile, polyetherimide, polysulfone, polyethersulfone and polyvinyliden fluoride form the porous support.

On this substructure a thin dense layer (in the range of 0.5–5 μm thick) is coated, which effects the separation. Different coating techniques are in use, most commonly a solution of the respective polymer in an appropriate solvent is spread onto the porous substructure. The solvent is then evaporated, followed by further treatment to effect cross-linking of the polymer.

In hydrophilic membranes the separating layer is made from cross-linked polyvinyl alcohol (PVA), from polyimides, or natural polymers such as chitosan or cellulose acetate (CA), with PVA dominant. For organophilic membranes, the separation layer is formed mostly from siloxanes such as polydimethylsiloxane (PDMS), or polyoctylmethyl siloxane (POMS).

In recent years new efforts have been made in academia and industry to develop new membranes for organic–organic separation. Of specific interest are the separation of olefins from paraffins, e.g. propene from propane, aromatics such as benzene or toluene from aliphatic hydrocarbons or the separation of the xylene isomers. To date, no industrialization has been achieved. The only industrial processes in this area are the separation of the light alcohols methanol and ethanol from their mixtures with hydrocarbons, ethers and esters. The membranes in use are, however, more of the hydrophilic type, in which the more polar alcohols replace the water.

To date, only polymeric membranes have been applied in pervaporation and vapour permeation processes. Thermal, mechanical and chemical stability of the porous substructure are limiting the operation range of this type of membrane, more than the stability of the separating layer. Demand for higher operation temperatures and chemical resistance have stimulated the development of inorganic substructures, and porous ceramics in particular. These can be coated by cross-linked polymeric separating layers similar to those on polymeric substructures. In more recent developments organic separation layers are applied, either by coating the porous substructure with a layer of zeolites or by reducing the size of the pore to molecular dimensions. The separation mechanism of these membranes is even more complex than that of polymeric separating layers, as molecular sieving effects, caused by shape and size of molecules, and molecule–surface interaction decide whether a component can pass through the membrane or will be retained.

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Modules

Design of modules for pervaporation and vapour permeation processes was based on the experience gained in water treatment by membranes, such as ultrafiltration and reverse osmosis processes. However, significant modifications had to be made because of the specific requirements of pervaporation and vapour permeation processes.

The partial vapour pressure at the permeate side has to be reduced in both processes to fairly low values, especially when low final concentrations have to be reached in the retentate. Therefore any pressure losses, even in the range of a few millibars have to be avoided at the permeate side. Since any feed mixture will contain organic components at high concentration, mostly at elevated temperatures, the chemical stability of all module components, such as spacer and potting material and glues is critical. To date, two types of modules are most widely applied:

- Plate modules, mainly used for dehydration applications, with permeate channels as open as applicable. Stainless steel is used as a construction material for support plates for the membranes and for spacers. The permeate channels are preferably open over the circumference of the modules which

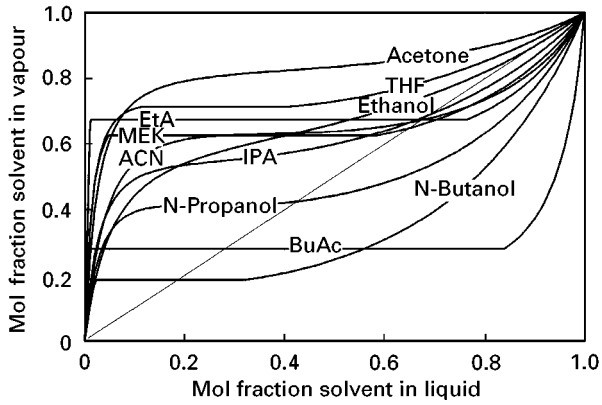

Figure 1 Vapour–liquid equilibrium curves for common pharmaceutical solvents which azeotrope with water. All can be dehydrated using pervaporation.

are assembled inside a special vacuum vessel that also house the permeate condenser. Alternative designs are very similar to plate heat exchangers, in which the supported membrane replace the heat exchanger plates. These modules are closed to the outside, with internal ducts feed and retentate, and for permeate removal.

- Spiral wound modules with stainless steel central tubes, but otherwise similar to those known from the conventional membrane processes, are mainly used for organophilic membranes. One or several of the spiral wound modules are housed inside a pressure tube and assembled in conventional skids. In a special design, the sandwich structures of membranes and permeate and feed spacer are welded together and not spirally wrapped around the central tube but arranged as flat sheets on the central tube for the removal of the permeate.

Very rarely, hollow fibres are used, generally with the feed flow inside the bore of the fibre. For the more conventional arrangement – feed flow at the shell side of the fibre – permeate pressure losses inside the bore may become detrimental for the process.

Applications

Organophilic Membranes

Organophilic membranes are mostly applied for the removal of volatile organic components (VOCs) from a gas stream such as waste air or nitrogen. The main applications are the treatment of streams originating from the evaporation of solvents in coating processes in film and tape production, purging of products such as polymers, by which unreacted monomers are removed, or from breathing of storage tanks for solvents, especially from loading and unloading of petrol tanks in tank farms. In many installations the feed stream received at atmospheric pressure is compressed in order to increase the feed side partial vapour pressure. Partial condensation of the component to be removed is a wanted side effect, since then condensation on the permeate side under vacuum and at low temperature can be avoided. The permeate is simply slightly compressed by the vacuum pump and let into the inlet of the feed compressor. In specific cases the installation of a vacuum pump will not be necessary and the permeate is obtained at atmospheric pressure.

The economy of the process is usually determined by the value of the components recovered. Emission regulations in all industrial countries demand for very low final concentrations if the gas stream is released to the atmosphere, therefore the retentate from the gas purification by the membrane is either recycled or followed by an additional polishing step.

Although considerable efforts in research and development have been devoted to the removal of

Table 1 Solvents routinely dehydrated using pervaporation/vapour permeation

Isopropanol, ethanol	Standard applications for pervaporation, typically dehydrated from their azeotropes to fractions of a percent of water. Many continuous, batch and vapour permeation units are operating around the world.
Ethyl acetate, butyl acetate	Form azeotropes in the miscibility gap and were traditionally dehydrated by two distillation columns and a phase separator, however with a massive recycle. Esters decompose in contact with zeolites. Pervaporation/vapour permeation is easily the best technique for dehydration.
Acetone	Does not azeotrope with water but when distilled a large reflux is required to get a half dry product. Pervaporation is ideal for final dehydration or for debottlenecking existing distillation systems.
Tetrahydrofuran	Easily dehydrated by pervaporation down to a few hundred ppm water. Traditional caustic washing is operationally messy, requiring a redistillation of the product. Pressure swing distillation requires high pressures and large recycles.
Methyl ethyl ketone	Pervaporation is again the preferred technique. Distillation is only possible with an entrainer because the azeotropic composition is nearly identical to the miscibility limit.
N-butanol, n-propanol	Form azeotropes with high water content so the distillation/phase separation process involves massive recycle streams. Pervaporation plants are less costly to build and easier to operate.

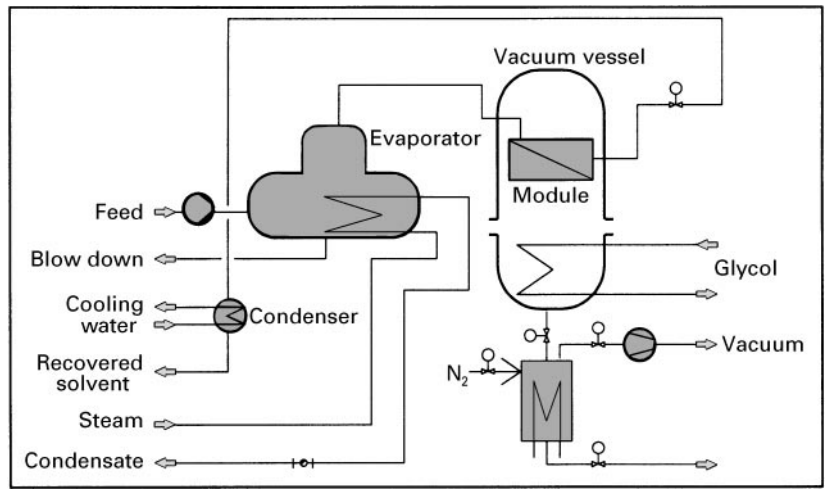

Figure 2 Typical flow diagram for recovery of solvent from mother liquors.

VOCs from aqueous stream, this technique has not yet been introduced into the industry. Potential mixtures which could be treated are more complex, the economical value of the recovered substances are low, and competing processes such as biological treatment of wastewater are cheaper. Applications may be found in the future in biotechnological processes where high-value products can be separated from a fermentation broth and be concentrated and purified in the same step.

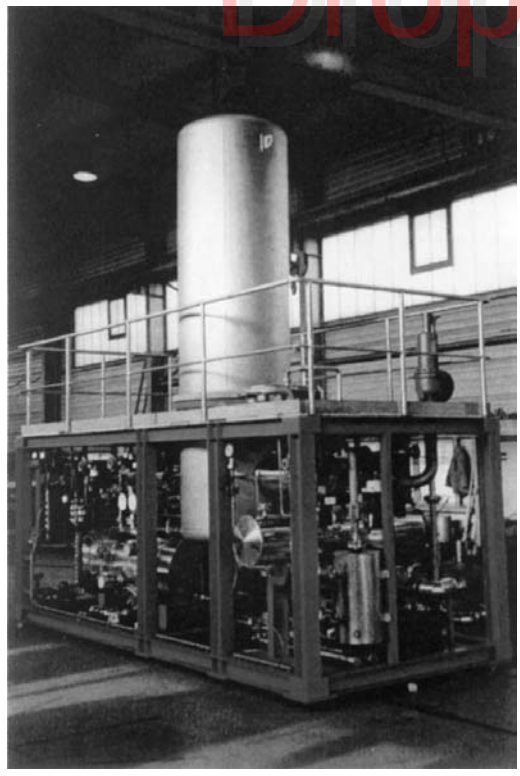

Figure 3 (See Colour Plate 51). Vapour permeation unit for recovering ink solvent.

Hydrophilic Membranes

The largest industrial installations of pervaporation and vapour permeation processes are equipped with hydrophilic membranes which are used for the removal of water from organic solvents and solvent mixtures.

Solvent dehydration Organic solvents are used for a variety of purposes in the chemical industry, e.g. for synthesis of pharmaceuticals, to precipitate materials from aqueous solutions, for cleaning purposes and for drying final products. Spent solvents nearly always contain some water. Dehydration is therefore an essential step in their recovery but difficult since most solvents from azeotropes with water. Final water removal by distillation is then impossible or complicated. Entrainer use is not an option for pharmaceutical or fine chemical production, where stringent process certification rules out adding potential sources of contamination.

Pervaporation enables solvents to be dehydrated without using any third substance or entrainer, simply, cheaply and without problems and irrespective of vapour/liquid equilibria (**Figure 1**). On-site solvent recovery using pervaporation and vapour permeation is thus becoming standard practice in the pharmaceutical and chemical industries (**Table 1**).

Often, pervaporation and vapour permeation plants are designed for operation with a number of solvents and with mixtures of solvents. Pervaporation and vapour permeation offers the following benefits when dehydrating solvents:

- No introduction of additional chemicals, complete solvent dehydration by pervaporation membranes irrespective of azeotrope formation and no possibility of contamination.

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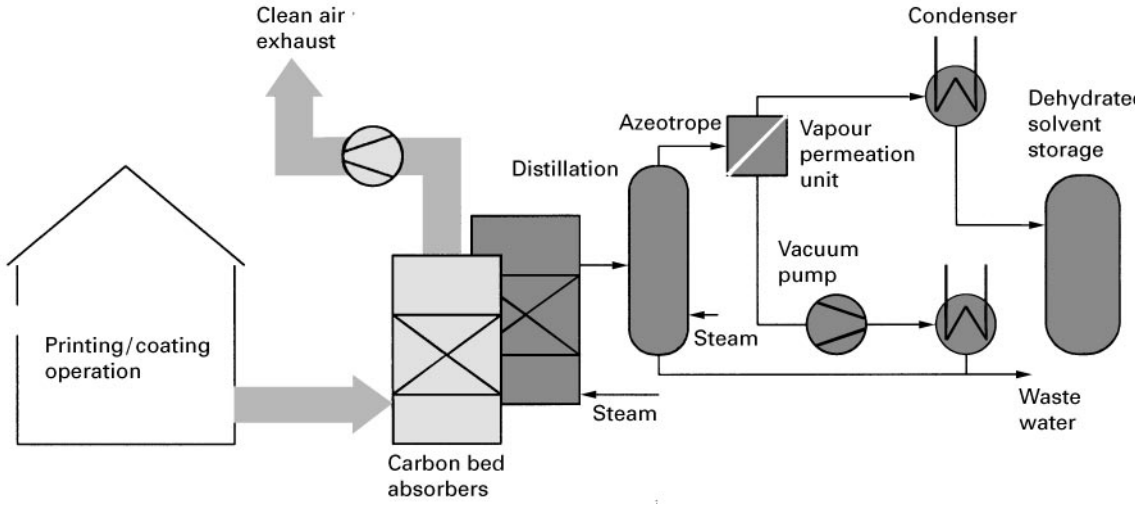

Figure 4 Carbon bed adsorption using steam regeneration and vapour permeation for final solvent dehydration.

- A choice of batch or continuous pervaporation systems, or continuous vapour permeation, depending on the duty.
- Able to dehydrate esters without any decomposition.
- Low energy consumption.

Solvent recovery from mother liquors Spent solvents (mother liquors) typically contain some water and are often saturated with dissolved material. They cannot be re-used without purification. Evaporation combined with vapour permeation is a powerful technique for purifying and dehydrating mother liquors (**Figure 2**).

The feed of spent solvent is evaporated and the resulting vapour is fed directly to a vapour permeation unit. Water vapour selectivity permeates the membrane and is condensed under vacuum. The water-free solvent vapour leaving the vapour permeation unit is condensed and is stored for re-use (product). A blowdown is taken from the evaporator to prevent buildup of dissolved solids. This purge can be treated to recover valuable components. Combining evaporation with vapour permeation gives the following benefits: only vapour is fed to the membranes – no possibility of fouling and no possibility of solids carryover into the recovered solvent; both the evaporation and the vapour permeation process steps are carried out in a single unit.

Solvent recovery from carbon bed adsorbers Biodegradable solvents such as alcohols and esters are used in many specific applications in coating and printing. Typically, a solution of the coating material is applied to the surface and the solvent is evaporated into an air stream, leaving a uniform film of coat material. The use of volatile solvents speeds the drying process.

The solvent-laden air stream cannot normally be discharged. It must be cleaned up and because the solvent loading is usually quite low, carbon bed adsorbers are most commonly used. These adsorbers are periodically regenerated, using either steam or nitrogen. Solvent is then recovered from the condensate

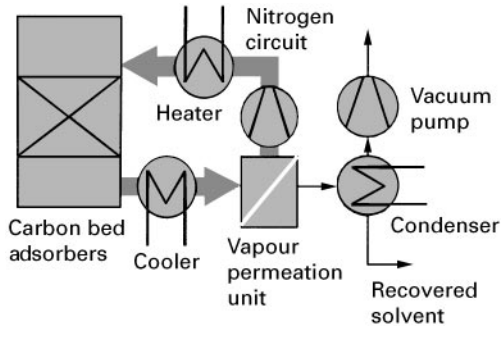

Figure 5 Use of vapour permeation to recover solvent from nitrogen desorption circuit.

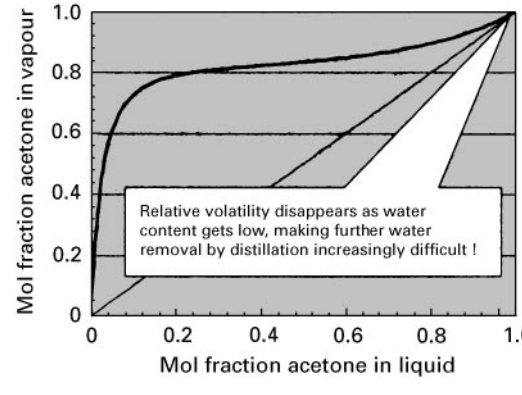

Figure 6 Vapour–liquid equilibrium diagram for acetone–water.

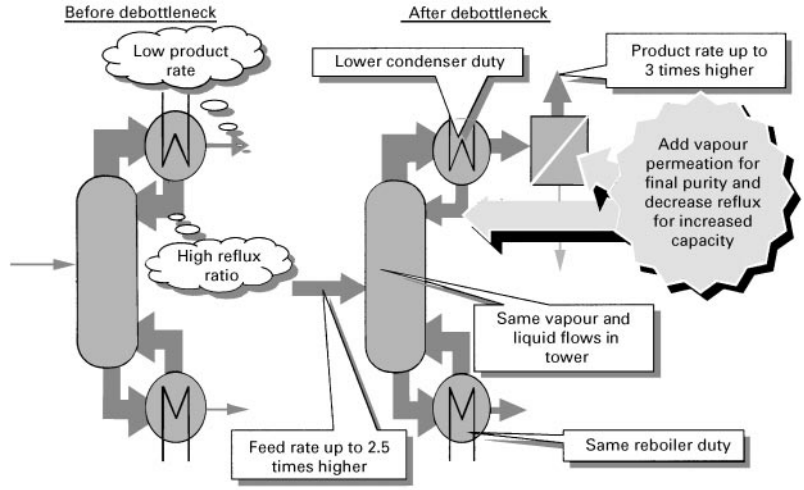
Before debottleneck
After debottleneck
Low product rate
Lower condenser duty
Product rate up to 3 times higher
Add vapour permeation for final purity and decrease reflux for increased capacity
High reflux ratio
Same vapour and liquid flows in tower
Feed rate up to 2.5 times higher
Same reboiler duty

Figure 7 Debottlenecking a single pinched column using pervaporation (acetone–water example).

or nitrogen stream for re-use in the coating/printing process (**Figure 3**).

To evaporate quickly, the recycled solvent has to be substantially dry and because most of the organics used form azeotropes with water, distillation is not sufficient for final dehydration. Pervaporation or vapour permeation provides a dry solvent at minimal cost.

Vapour permeation for solvent dehydration in printing and coating If carbon bed adsorbers are regenerated with steam, the condensate is typically steam distilled up to the azeotrope (**Figure 4**). Continuous coating operations use continuous distillation to concentrate condensate from the bed regeneration. A vapour permeation system is normally connected directly to dehydrate net overhead vapour from the distillation. This situation is shown schematically below. In this case, no additional energy is required for the final dehydration by vapour permeation.

Vapour permeation for solvent removal from circulating nitrogen If nitrogen is used to regenerate the carbon bed, solvent vapour can be continuously removed by vapour permeation through an organophilic membrane. This is much more economical than cooling the vapour to condense the solvent; because the solvent loading is low (**Figure 5**).

The use of vapour permeation for solvent recovery in printing and coating operations provides the following benefits: ethanol, isopropanol, ethyl acetate and other biodegradable solvents are recovered and dehydrated without entrainers and economically,

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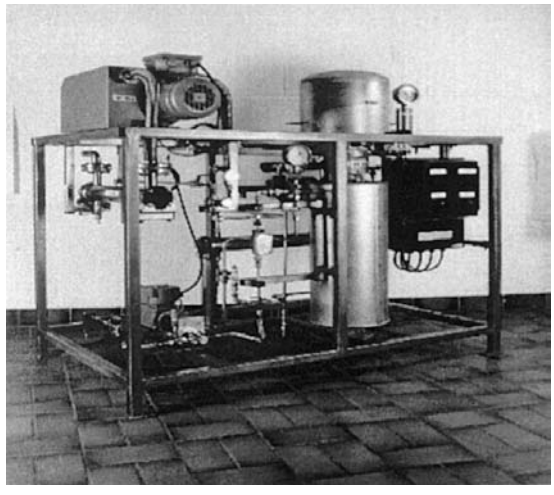

Figure 8 (See Colour Plate 52). Standard unit for batch dehydration of rinse alcohol.

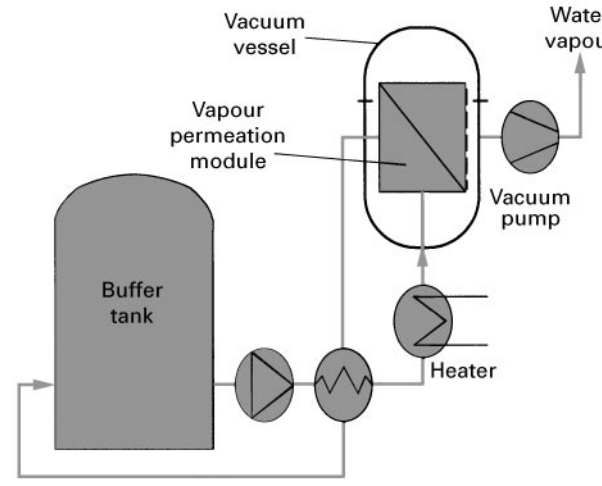

Figure 9 Batch pervaporation process for dehydration of rinse alcohol.

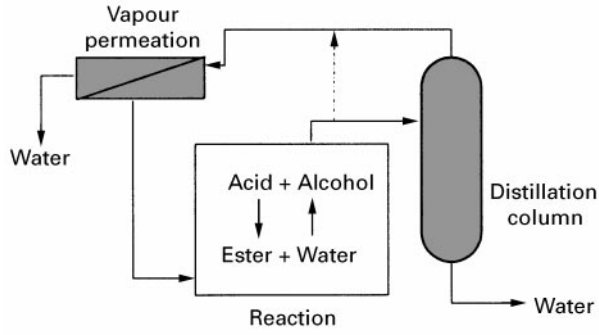

Figure 10 Water removal by distillation and vapour permeation.

even at a modest scale; solvent can be economically recovered from nitrogen streams with minimal cooling requirement.

Debottlenecking Distillations

Debottlenecking pinched distillations Distillation processes are driven by volatility differences. If these volatility differences are small, or become small under certain conditions, then columns need to operate with high reflux to achieve the desired separation. Because pervaporation/vapour permeation processes separate irrespective of volatility differences, they can be used very effectively to debottleneck pinched distillations.

Consider for, example, the system acetone–water (**Figures 6** and **7**). Acetone is concentrated in the vapour phase at low concentrations so stripping of acetone from water is easy. At high concentrations this is not the case. Complete dehydration of acetone is difficult.

Debottlenecking entrainer distillation systems Existing entrainer distillation systems can also be effectively debottelenecked using pervaporation/vapour permeation. Normally, the rectification column will be operating to give a product as close to the azeotrope as possible, running with a high reflux. To debottleneck the system, reflux in the rectification column is reduced, giving more overhead product, but with a higher water content. The pervaporation unit is sized to remove enough water that the subsequent entrainer column is also unloaded. Both columns can then realize a significant capacity increase.

The pervaporation unit required for debottlenecking is relatively small since the driving force for water permeation is high. Adding a pervaporation/vapour permeation system to a pinched distillation can give providing higher product capacity and reduced re-

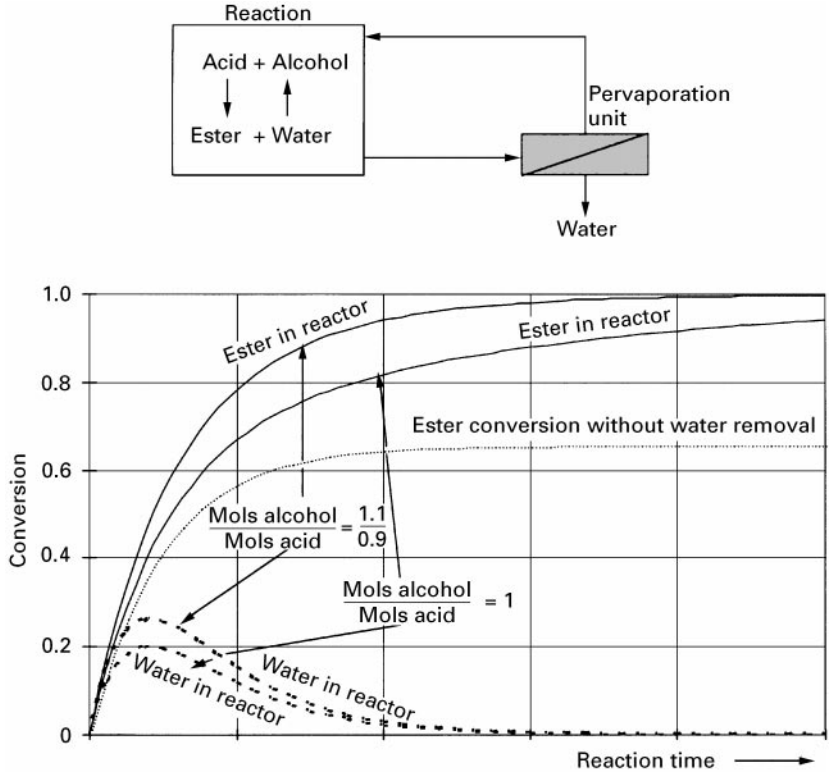

Figure 11 Progression of a batch esterification (with equilibrium constant $K = 4$) with continuous water removal by pervaporation.

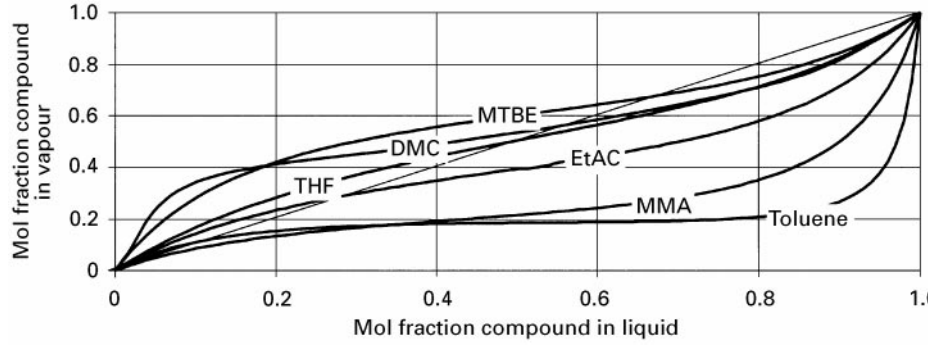

Figure 12 Some methanol azeotropes which can be separated using pervaporation vapour permeation.

flux, significantly higher products purity and reduced energy costs.

Dehydration and Purification of Rinse Alcohol

Many metal components used in the electronics industry undergo a rinse process using ethanol or isopropanol. Typically, the surface is first treated in another way to remove contaminants and then washed with water. The final rinse with alcohol displaces the water and any remaining contaminants and also wets the surface completely. The volatile alcohol then dries uniformly leaving a clean unmarked surface. Because the rinse alcohol displaces water it gradually becomes diluted and loses its drying qualities. Either fresh alcohol must be purchased or the alcohol must be dehydrated and purified to restore its performance.

The purity of the rinse alcohol is critical for component performance; wafer fabrication, for example, sets p.p.b. limits on certain metal ions (**Figure 8**).

Various standardized pervaporation/vapour permeation systems are now used economically and at a site scale for dehydration and purification of rinse alcohol. A batch of used alcohol is continuously circulated from a buffer tank via a recuperator, heater and pervaporation module and water vapour is continuously removed via the vacuum pump (**Figure 9**). The batch is processed until the required degree of dryness is reached. Standardized units economically treat batches as small as 1 m^3 day^{-1}.

Use of pervaporation and vapour permeation units to recover rinse alcohol gives the following benefits: minimal alcohol losses, very high product purities can be reached, and economical recovery at site scale.

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Continuous Water Removal from Condensation Reactions

Condensation reactions such as esterifications, acetalizations and ketalizations produce water as coproduct; removal of this water from the reaction will shift the equilibrium in favour of the desired product.

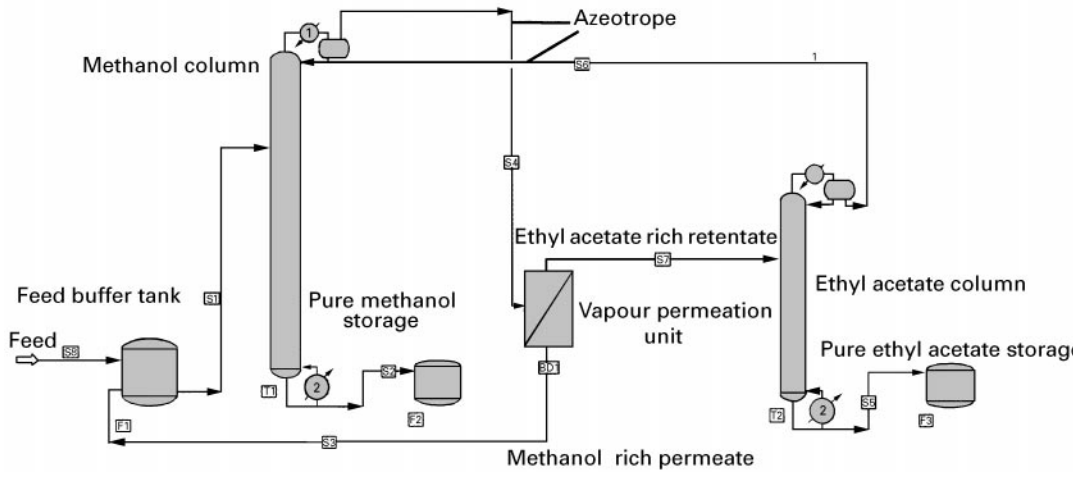

Figure 13 Methanol recovery by azeotrope breaking (methanol–ethyl acetate example).

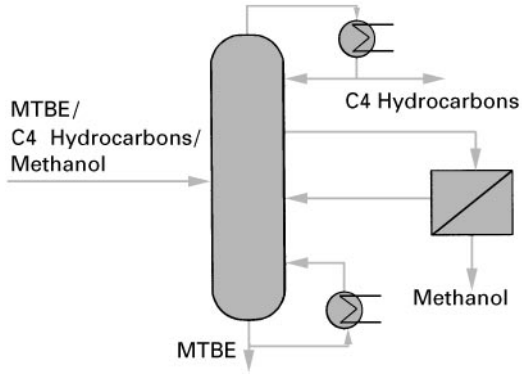

Figure 14 Methanol removal by pervaporation of column side-draw.

Distillation is often used to remove water from condensation reactions. However complete water removal is difficult because alcohols, esters and acids typically azeotrope with water. Boiling the reaction mix also removes alcohol, which is normally the most volatile component including a vapour permeation unit after the distillation step avoids the problems of azeotrope formation. Water can be completely removed (**Figure 10**).

Water removal by pervaporation only – membrane reactors Removing water directly from the reaction mix is more effective – the reaction can even be run under stoichiometric conditions. Reactor configuration is simpler and energy consumption is much lower (**Figure 11**).

Using pervaporation/vapour permeation units to continuously remove water from condensation reactions gives the following benefits: complete conversion, maximum yield, minimum reagent consumption and costs; maximizes reaction kinetics, reactor efficiency and productivity; minimizes product purification costs; works irrespective of azeotrope formation.

Methanol Recovery

Methanol is commonly used as both solvent and reactant in the chemical industry. However, it forms azeotropes with many substances, particularly esters (**Figure 12**). Methanol often cannot be removed from spent solvents or from reaction mixtures with simple distillation. Some quite complicated processes have been developed to get around this problem.

Industrial pervaporation/vapour permeation units are now used for separation of methanol, either stand alone or in combination with distillation.

Methanol recovery by azeotrope breaking By way of example, a separation scheme for a methanol-rich methanol–ethyl acetate mixture is shown below. The mixture is distilled to the azeotrope, taking pure methanol out as bottom product. The overhead stream is passed directly to a vapour permeation unit which permeates a methanol-rich stream. This stream is condensed and passed back to the methanol column via the feed buffer. Retentate from the vapour permeation unit, strongly depleted in methanol, can be fed directly to the ethyl acetate column. Pure ethyl acetate leaves this column as bottom product while overhead azeotrope is sent to the vapour permeation unit (**Figure 13**).

Many solvent or ester/methanol mixtures can be separated using a similar scheme. If the feed is close to the azeotrope then the methanol column can be dispensed with. If the capacity is small the purification column for the second component may not be required, depending on the desired purity.

Institut Français du Pétrole (IFP) has developed a process where pervaporation of methanol is used to debottleneck MTBE production. In the debutanizer columns used in MTBE processing, the MTBE–methanol azeotrope results in a concentration of methanol at a point midway between the feed tray and the reboiler (**Figure 14**). Pervaporating methanol out of the process from a side-draw taken at this point results in methanol free MTBE as debutanizer bottom product.

Separation systems based on pervaporation/vapour permeation of methanol offer the following benefits: problem-free separation of methanol/organic mixtures irrespective of azeotrope formation; avoids water wash for methanol removal; minimum energy costs.

See Colour Plates 51, 52.

Further Reading

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